Purpose

The purpose of this procedure is to describe the setup, measurement and reporting procedures for the absorbed-dose certification of customer-irradiated NIST transfer dosimeters.

Scope

NIST provides transfer standards in the form of sets of calibrated alanine dosimeters packaged in polystyrene. The sealed, packaged dosimeters are sent to the customer for irradiation to nominal agreed–upon absorbed dose levels in a prescribed geometrical arrangement. The unopened packaged dosimeters are then returned to NIST to be measured and evaluated and the results reported in the form of an absorbed-dose certificate. The absorbed dose range that is suitable for use with the transfer dosimeters is 50 Gy to 200 kGy.

Definitions

Absorbed dose to water: the energy absorbed from ionizing radiation per unit mass of water: 1 J/kg = 1 Gy.

Dosimeter batch: quantity of dosimeters made from a specific mass of material with uniform composition fabricated in a single production run under controlled, consistent conditions, and having a unique identification code.

Electron Paramagnetic Resonance (EPR): the process of resonant absorption of microwave radiation by paramagnetic ions or molecules, with at least one unpaired electron spin, and in the presence of a static magnetic field.

Single-Hole vial geometry (SH): irradiation geometry with pellets stacked vertically in a polystyrene vial that is placed in the absolute center of the isodose irradiator-source profile. This geometry is used as the calibration point to which all other geometries are referenced.

Equipment

Essential Equipment	**Calibration Method**	**Calibration Frequency**
Cobalt-60 Pool Source	Comparison to Vertical Beam Source	Determined by control charts
MDS Nordion Gammacell 45	Comparison to Pool Source	Annual
MDS Nordion Gammacell 232	Comparison to Pool Source	Annual

Essential Equipment	**Calibration Method**	**Calibration Frequency**
MDS Nordion Gammacell 207	Comparison to Pool Source	Annual
Bruker EMS 104 EPR Spectrometer	Dosimeter Check Dose Measurement	As needed
Bruker ECS 106 EPR Spectrometer	Dosimeter Check Dose Measurement	As needed
Denver Instrument Company Microbalance, Model A-160	External Service	Annual
Platinum Thermometer	External Service	Annual
Type-T Thermocouple	Comparison to Platinum Thermometer	Determined by control charts
Non-Essential Equipment		**Calibration Frequency**
Mitutoyo, Mini-checker Digital Electronic Gauge		As needed

Health & Safety Precautions

Radiation safety
Rooms containing ^{60}Co sources have been designated as High Radiation Areas. Radiation safety and training services are provided by the NIST Health Physics Office.

Magnetic field safety
People with pacemakers should avoid rooms containing electromagnets associated with EPR spectrometers.

Procedures[†]

1. *Dosimeter Batch evaluation and testing*

1.1 General

1.1.1 This procedure describes the requirements for performance characterization of transfer dosimeters used in NIST high-dose dosimetry certification services. It is intended to provide data that identifies influence quantities that may have significant effects on the performance of a dosimetry system.

1.1.2 This procedure is intended for routine evaluation of a commercially available dosimetry system. Evaluation of a new

[†] The mention of commercial products throughout this paper does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that products identified are necessarily the best available for this purpose.

dosimetry system in the development phase or the evaluation of a new commercial dosimetry system may require additional tests or more extensive testing not described here.

1.1.3 Irradiation and measurement procedures are described in Sections 3 and 4, respectively. Irradiations should be done at ambient temperature, unless stated otherwise.

1.1.4 Data from all tests described in this section shall be recorded in the Dosimetry System Databook. A summary of a history of these evaluations should be an integral part of the compilation.

1.1.5 Acceptance criteria for the tests described in this section are listed in Appendix A. A failure to meet these criteria shall result in a repeat of the specific test(s) that failed. Continued failures must be resolved by either correcting or controlling the influence quantity. If corrective action is required, see IRD Guide IRD-G-08.

1.1.6 Reasonable efforts should be made to minimize the exposure of stored dosimeters to environmental influences despite data that indicate exposure of commercial alanine dosimeters to moderate variations in ambient temperature, relative humidity, and light pose no long-term problem.

1.2 Selection

1.2.1 Determine the total number of dosimeters needed for the performance characterization.

1.2.2 The selection of test dosimeters should attempt to be random, both in creating the working subset of dosimeters (1.2.3 below) and in selecting from this subset of dosimeters to perform specific tests.

1.2.3 If the dosimeter batch is distributed over several containers, the selection process should include dosimeters from each container to the extent practical. Containers should possess unique identifiers.

1.2.4 If the individual dosimeters do not possess unique identifiers, they should be marked in such a manner that uniquely identifies them.

1.2.5 Records should include a correlation between the dosimeter identifier, its batch, and the container from which it was removed.

1.3 Mass test

1.3.1 This test is only required for alanine pellet dosimeters; it is not applicable to alanine film dosimeters.

1.3.2 Select and weigh dosimeters to the nearest 0.0001 g.

1.3.3 Plot a histogram of the mass distribution.

1.3.4 Determine the mean, standard deviation, and relative standard deviation of the dosimeters measured.

1.4 Height test

1.4.1 This test is only required for alanine pellet dosimeters; it is not applicable to alanine film dosimeters.

1.4.2 Select and measure the dosimeter heights to the nearest 0.01 mm.

1.4.3 Plot a histogram of the height distribution.

1.4.4 Determine the mean, standard deviation, and relative standard deviation of the dosimeters measured.

1.5 Response variation

1.5.1 Irradiate 4 dosimeters to each of the prescribed doses: 0.10, 1.0, 10, and 100 kGy for alanine pellets, and 1.0, 10, 100 kGy for alanine film.

1.5.2 Measure the EPR response (see Section 4) of the individual dosimeters.

1.5.3 Determine the mean, standard deviation, and relative standard deviation of the measured dosimeters grouped by prescribed absorbed dose.

1.6 Dose fractionation

1.6.1 Irradiate (film or pellet) dosimeters to prescribed doses.

1.6.1.1 Irradiate six dosimeters (groups 3A & 3B) to 1.0 kGy, wait 1 hour then irradiate the six dosimeters to 1.0 kGy, remove three dosimeters (group 3B), wait 1 hour then irradiate the group 3A dosimeters to 1 kGy, wait overnight (~20 hours), then irradiate group 3B to 1.0 kGy.

1.6.1.2 Irradiate three dosimeters (group 3C) to 3.0 kGy.

1.6.1.3 Irradiate six dosimeters (groups 30A & 30B) to 1.0 kGy, wait 1 hour then irradiate the six dosimeters to 1.0 kGy, remove three dosimeters (group 30B), wait 1 hour then irradiate the group 30A dosimeters to 1 kGy, wait overnight (~20 hours), then irradiate group 30B to 1.0 kGy.

1.6.1.4 Irradiate three dosimeters (group 30C) to 30 kGy.

1.6.2 After a 24 h wait period measure the absorbed dose for all dosimeters.

1.6.3 Determine the mean, standard deviation, and relative standard deviation of the dosimeter groups measured and compare fractionated doses to continuously applied doses.

1.7 Relative humidity (%RH)

1.7.1 Store 12 (film or pellet) dosimeters in controlled 0 %RH and 55 %RH environments [1].

1.7.2 After ~4 days remove dosimeters and quickly seal dosimeters in groups of three in a sealed vial (pellets) or sealable sachet (film).

1.7.3 Irradiate a pair of 0 %RH and 55 %RH dosimeters to 1.0 kGy.

1.7.4 Irradiate a pair of 0 %RH and 55 %RH dosimeters to 10 kGy.
1.7.5 Allow dosimeters to equilibrate with the laboratory ambient %RH for 1 to 2 hours before measuring the response for each dosimeter.
1.7.6 Determine the mean, standard deviation, and relative standard deviation of the dosimeter groups measured and compare by absorbed dose delivered and %RH.

1.8 Post-irradiation time dependence
1.8.1 Irradiate six (film or pellet) dosimeters, three to 1.0 kGy and three to 10 kGy.
1.8.2 Measure the absorbed dose approximately every other day for 2 weeks.
1.8.3 Determine the mean, standard deviation, and relative standard deviation of the measured dosimeters according to absorbed dose delivered.

1.9 Irradiation temperature coefficient
1.9.1 In the disc-shaped aluminum irradiation geometry, irradiate (film or pellet) dosimeters to 10 kGy at each of the three irradiation temperatures: ambient; ambient + 20 °C; and ambient - 20 °C.
1.9.2 Measure the EPR response of the individual dosimeters.
1.9.3 Compute the resultant temperature coefficient (slope of the percent change, relative to the predicted value at the ambient temperature, in response versus the irradiation temperature) and compare this value to the accepted value.

1.10 Batch mean
1.10.1 Irradiate simultaneously (co-located) two dosimeters from previous batch with two dosimeters from the batch undergoing evaluation to each of the following doses: 1.0 kGy and 10 kGy.
1.10.2 Measure the EPR response (not absorbed dose) of the individual dosimeters.
1.10.3 Determine the mean of the dosimeter groups and compute the percent difference in response between the batches.

2. *Instrument maintenance*

2.1 ECS106 EPR Spectrometer
2.1.1 As necessary, or at least every six months, the in-line filter for the cooling water must be replaced.
2.1.2 As necessary, or before each new calibration curve is measured, the quartz sample tube should be cleaned.
2.1.2.1 Place a ~1 liter beaker under sample cavity area.
2.1.2.2 Flush tube with ~ 200 ml acetone.

2.1.2.3 Flush tube with ~ 200 ml ethanol.
2.1.2.4 Flush tube with ~ 200 ml methanol.
2.1.2.5 Draw a vacuum until sample tube is dry (as evidenced by the return of a symmetric tune mode pattern to the spectrometer display).
2.1.3 Record all maintenance activities in the ECS106 maintenance log book.

2.2 EMS104 EPR Spectrometer
2.2.1 Spectrometer should remain on continuously. In the event of a prolonged power outage, power-up instructions reside in the EMS104 log book.

3. ***Dosimeter batch calibration***

Once a batch of dosimeters has been characterized and passed all acceptance criteria, a calibration curve shall be established.

3.1 Initiate irradiation data record
3.1.1 Refer to the Irradiation Facilities Record Book to get the dose rate for the chosen irradiator with the single-hole vial geometry.
3.1.2 Open the excel spreadsheet databook template and enter the appropriate header information, including the above mentioned annual dose rate value.
3.1.3 Before each irradiation, enter an approximate value into the "start time" cell so the program can calculate the decay, dose rate, and irradiation time.

3.2 Prepare dosimeters for irradiation
3.2.1 Alanine pellet dosimeters:
3.2.1.1 Fill a single hole vial with pellets. Depending on the pellet height, it will accept 3 or 4 pellets.
3.2.1.2 Choose an appropriate length polystyrene stem according to the Irradiation Facilities record book.
3.2.1.3 Screw an aluminum base onto the stem.
3.2.1.4 With a foam collar on the stem, place the single-hole vial into the collar to hold it atop the stem.

3.2.2 Alanine film dosimeters:
3.2.2.1 Choose an appropriate length polystyrene stem according to the Irradiation Facilities record book.
3.2.2.2 Screw an aluminum base onto the stem.
3.2.2.3 Secure a 12mm polystyrene sleeve atop the stem with electrical tape.
3.2.2.4 Drop four films into the open sleeve. The films don't

require any packaging.

3.3 Control dosimeter temperature

3.3.1 Dosimeters are normally irradiated at room temperature, 22-25^{o}C. GC45 does not require cooling to maintain ambient temperature, but to maintain that temperature in GC232 or GC207 requires blowing compressed air into the GC sample chamber. To do this, open the valve of the flow meter mounted on the side of the appropriate Gammacell. Temperature is monitored with a type-T thermocouple placed inside the GC sample chamber. On the computer, start the temperature recording program.

3.3.2 The type-T thermocouple is calibrated against the high-precision platinum thermometer in the GammaCell sample chamber (in the up position) over a temperature range that corresponds to service irradiations. The operational status of the thermocouple is monitored by periodic checks and control charts. Thermocouples that do not perform within the control limits are replaced.

3.4 Operate Gammacell

3.4.1 Place dosimeter assembly into the GC sample chamber.

3.4.2 Set timer to desired irradiation time, as calculated by the databook spreadsheet.

3.4.3 Press the “DOWN”(GC45) or “CYCLE START”(GC232) or “START” (GC207) button to begin the irradiation.

3.4.4 When the chamber arrives at the down position, note the actual clock time and enter it into the databook spreadsheet.

3.4.5 When the timer finishes and GC sample chamber comes up, remove the dosimeters.

3.5 Record irradiation data

3.5.1 After each irradiation, use the computer program to calculate the average temperature during irradiation. Enter the value into the databook spreadsheet.

3.5.2 When all irradiations are finished, save the file, print the spreadsheet and paste it into the High-Dose Irradiations Databook.

3.6 Measure dosimeters

3.6.1 After all irradiations are completed, measure the dosimeters as described in Section 4.

3.7 Analyze data

3.7.1 Enter dose and measured response values into the curve fitting software, TableCurve2D (made by Systat Software Inc.). Have the software fit the four usual equations: Linear, 3rd order polynomial,

Exponential saturation (as defined in the Dosimetry System Databook), Exponential saturation with forced origin.

3.7.2 Choose the best fit, based on experience, residuals analysis, and F-statistic.

3.8 Record calibration curve

3.8.1 Print the graph of the fitted equation and the graph of the residuals (in percent).

3.8.2 Paste the two graphs into the Dosimetry System databook.

3.8.3 Type the new curve coefficients into the appropriate formulas of the excel spreadsheets that calculate dose from dosimeter response.

4. *Customer-irradiated absorbed-dose certification for alanine pellets*

4.1 Customer contact

4.1.1 Receive Purchase Order via mail or fax

4.1.2 Input customer information into calibration log book, and assign a Division calibration number (HDxxxx)

4.1.3 Mail out dosimeters (see Section 6.2) for requested test (service) numbers with instruction letter to customer (Appendix B)

4.1.3.1 Each dosimeter consists of four alanine pellets in a polystyrene vial [2], or four alanine films folded into thirds and sealed in an aluminized pouch.

4.1.4 Input PO and customer information into Information System to Support Calibrations (ISSC) database to be assigned a folder number

4.1.5 After folder number is assigned, print fee sheet and division record

4.1.6 Fax PO and folder number assignment to Measurement Services Division

4.1.7 Receive calibration test folder via NIST internal mail, and insert PO, fee sheet, and division record

4.2 Receiving customer dosimeters

4.2.1 Verify that irradiator and irradiation information (target dose, temperature, etc) on instruction letter has been provided by customer

4.2.2 Turn on the ECS106 Spectrometer (see Section 4.3)

4.2.3 Remove dosimeters from vials, numbering consecutively (top to bottom) as removed from the vial

4.2.4 Empty vials are cleaned by immersing in ethanol with agitation, allowing them to remain immersed overnight, then dried in a fume hood overnight

4.2.5 Weigh pellets (see Section 4.4) and input masses into excel spreadsheet

4.2.6 Create excel spreadsheet (see Section 4.5) to record data from measurements

4.3 ECS106 setup

4.3.1 Turn on chilled water supply to midpoint of valve setting (if magnet is not cool to the touch during operation, increase the water flow)

4.3.2 Turn on magnet

4.3.3 Turn on the ECS106 computer component

4.3.4 Logon to the ECS106, using the applicable login name (refer to ECS106 Maintenance log book)

4.3.5 Once the standby light changes from red to green, use the right arrow key to change to tune mode

4.3.6 With an unirradiated dosimeter in the EPR sample tube, use the up-arrow key to perform the auto-tune procedure at least twice, and allow approximately one hour for the spectrometer to warm up, then run the auto-tune procedure once more

4.3.7 On page 0, load the spectral parameters that were used for the corresponding calibration curve (refer to Dosimetry System Databook)

4.3.8 On page 1, load the ruby parameters

4.3.9 On page 0, load the applicable measurement routine command file (see Appendix C)

4.3.10 Set the parameters to the instrument by scrolling to the Parameters list and clicking the 'greater than' (>) sign

4.4 Mass determination

4.4.1 Access the dosimeter mass measurement program from a computer local to the analytical microbalance

4.4.2 Enter wait time for tare and mass stabilization, typically 7-9 seconds

4.4.3 Press enter to start transmission or Q to quit

4.4.4 Enter the starting pellet number

4.4.5 Weigh and remove pellets as program prompts

4.4.6 Input pellet masses to the measurement spreadsheet

4.4.7 Press any key to stop transmission, then Q to quit

4.5 Excel spreadsheet setup

4.5.1 Verify that the Parameter worksheet contains the ECS106 parameters matching those of the corresponding calibration file (refer to Dosimetry System Databook)

4.5.2 On the Xferdata worksheet, create columns for the date, vial number, target dose, pellet mass, alanine signal max/min peak height and ruby signal max/min peak height (for each orientation)

4.5.3 Set Excel worksheet to automatically calculate Peak-to-peak amplitude, mass-normalized and ruby-normalized signal values

4.5.4 On the Summary worksheet, create a location for the company name, input the name, date, filename, and coefficients of the calibration curve being used, the calculated ruby/mass normalized signal (from the Xferdata worksheet), the calibration temperature and temperature coefficient, and the irradiation temperature (from the customer instruction letter)

4.5.5 Set Excel worksheet to automatically calculate the temperature corrected response and dose

4.6 ECS106 measurement

4.6.1 Using the in-house vacuum line with suction attachment, insert dosimeter ensuring that the instrument remains tuned (meters in the spectrometer display should be at the midpoint, else the spectrometer should be retuned).

4.6.2 Run the measurement procedure by clicking A (Acquisition), then E (Execute) on the drop down menu

4.6.3 Rotate the sample tube according to the markings of the guide mounted to the cavity (~ 120°)

4.6.4 Run the measurement procedure again

4.6.5 Using the in-house vacuum line with suction attachment, remove dosimeter

4.6.6 Repeat steps 1 through 5 for each dosimeter to be measured

4.6.7 After the last dosimeter has been measured, scroll down to page 0

4.6.8 Press D (Data Handling), D (Peak Picking) on the drop down menu, M (Find Extremal Values)to select the maximum and minimum peak-to-peak values

4.6.9 Input each maximum and minimum (absolute) value into columns designated 1st orientation alanine signal, ruby signal, and 2nd orientation alanine signal, ruby signal

4.6.10 Use the ↑ key to navigate through the pages until all data is recorded

4.7 Analysis

4.7.1 Check data for anomalies by paying close attention to the RSD for each measurement (ruby and pellet), perform outlier test and/or repeat measurement as needed

4.7.2 Calculate dose by using the coefficients from the applicable calibration curve (refer to Dosimetry System Databook)

4.7.3 Make sure that data has been reviewed by the proper supervisor

4.7.4 Print copies of data, and insert in Transfer Dosimetry Databook, as well as calibration folder

4.7.5 Copy Transfer Dosimetry Databook pages and insert into HD folder

4.8 Report and test closure

4.8.1 Complete the QM Checklist for 49020C and 49030C (Appendix D) to ensure proper completion of the data analysis procedure

4.8.2 Create an "Absorbed Dose Measurement Certificate" report by inserting the company information, folder number and Transfer Dosimetry Databook references (footnote), and the final dose values into the report template (see Appendix E)

4.8.3 Log in to the ISSC database

4.8.4 Select "Navigational Tools"

4.8.5 Type the folder number into the "View shortcuts for FN" prompt

4.8.6 Select the Close test and enter data link

4.8.7 Select test to be closed from the list provided

4.8.8 Verify the calibration date is accurate

4.8.9 Select the close status

4.8.10 Note the Transfer Dosimetry Databook page numbers in the Notebook Reference box

4.8.11 Select "Submit values"

4.8.12 Select "Do not print calibration report"

4.8.13 Sign Fee sheet and Division Record (DO NOT DATE – will be dated by Administrative Officer)

4.8.14 Make 2 photocopies of the Fee sheet, and 1 photocopy of the Division Record

4.8.15 Staple original Fee sheet and one photocopy of Fee sheet together

4.8.16 Paperclip original Division record to stapled Fee sheets

4.8.17 Sign blue test folder, and check the 3 year box

4.8.18 Staple the contents of the test folder, with a photocopy of the calibration report on top, to the inside back cover of the test folder

4.8.19 Staple photocopy of the Fee sheet to the inside front cover of the test folder

4.8.20 Staple photocopy of the Division Record to the inside front cover of the HD folder

4.8.21 Paperclip copies to the front of the test folder and place folder in AO's inbox

4.8.22 Write "Mailed on" date on photocopy of calibration report and place in HD folder

5. Customer-irradiated absorbed-dose certification for alanine films

5.1 Customer contact procedure same as for alanine pellets

5.2 Receiving customer dosimeters

5.2.1 Verify that irradiator and irradiation information (target dose, temperature, etc) on instruction letter has been provided by customer

5.2.2 Remove dosimeters from packaging, recording the correlating film and package numbers into the spreadsheet

5.2.3 Create Excel spreadsheet (see Section 4.5) to record data from measurements

5.3 EMS104 setup

5.3.1 Spectrometer should not be turned off; in the event of a prolonged power outage, refer to EMS104 log book for setup from power off position

5.3.2 Set parameters according to those that were used for the corresponding calibration curve (refer to Dosimetry System Databook)

5.3.3 Insert and orient film sample tube by making repeated measurements to achieve the maximum signal from an irradiated test film, then tighten the collet to fix the tube position.

5.4 Excel spreadsheet setup

5.4.1 Verify that the Excel parameter worksheet contains the EMS104 parameters matching those of the corresponding calibration file (refer to Dosimetry System Databook)

5.4.2 On the Xferdata worksheet, create columns for the date, film number, target dose, signal amplitude

5.4.3 On the Summary worksheet, input the company name, input the name, date, filename, and coefficients of the calibration curve being used, the signal amplitude (from the Xferdata worksheet), the irradiation temperature (from the customer instruction letter), the calibration temperature, and the temperature coefficient.

5.4.4 Set Excel worksheet to automatically calculate the temperature corrected response and dose.

5.5 EMS104 measurement

5.5.1 Set parameters according to the Excel parameter worksheet values.

5.5.2 Insert film into the sample tube

5.5.3 Press Acquire button

5.5.4 Record signal amplitude into excel worksheet

5.5.5 Repeat steps 1 through 3 for calibration curve film first, then for each test dosimeter to be measured

5.6 Analysis

5.6.1 Check data for anomalies, perform outlier test and/or repeat measurement as needed
5.6.2 Calculate dose by using the coefficients from the applicable calibration curve (refer to Dosimetry System Databook)
5.6.3 Make sure that data has been reviewed by the proper supervisor
5.6.4 Print copies of data, and insert in Transfer Dosimetry Databook, as well as calibration folder
5.6.5 Copy Transfer Dosimetry Databook pages and insert into HD folder

5.7 Report and test closure procedure same as for alanine pellets

6. Quality control

6.1 Absorbed-dose check standards

Dosimeter pellets are irradiated to each of the following doses: 1, 10, 50 kGy. These check standards are routinely measured ~48 hours after irradiation, as well as measured prior to service measurements that were irradiated close to the same date. Once each week for two more weeks, these dosimeters are measured. Data from these check standards are compiled into a control chart for tracking and comparison in the Quality Control Databook. At the end of three weeks, the process is repeated. Check standards for doses outside of the 1-50 kGy range are generated and measured as needed. Film check standards are produced and measured as needed. Check dose measurements that measure outside of set limits must be resolved through re-measurement, repetition of the check standard process, or reconfiguration of spectrometer settings (the latter may require a total recalibration of the dosimetry system).

6.2 Transfer dosimetry controls

Each set of transfer dosimeters shipped to a customer is paired with a control vial that is packaged separately and marked “Do Not Irradiate”. Control vials contain dosimeters of the same type that have been previously irradiated to a calibrated dose. These dosimeters are typically check standards that have exhibited good stability after repeated measurements. A continuous history of these data is recorded in the Quality Control Databook. Any nonconformance shall be reported; action to be taken is at the discretion of the calibration staff.

6.3 International Comparisons

Approximately annually, dosimetry comparisons are performed with the high-dose calibration facility of the National Physical Laboratory of the United Kingdom. Dosimeters from each facility are exchanged, measured, and the results compared. Participation in larger international comparisons

occur when appropriate. These data are summarized in the High-Dose International Comparisons Databook.

7.Traceability

7.1 The SI unit of absorbed dose is the Gray (Gy). For this service, the Gray is realized through water calorimetry measurements in the Vertical Beam Cobalt-60 Gamma-Ray Source. These measurements are transferred to the Pool Source and subsequently to the three GammaCell calibration sources by source-rate ratio measurements using alanine dosimetry. These transfer measurement protocols are described in NIST SP250-44 (See: http://ts.nist.gov/ts/htdocs/230/233/calibrations/Publications/series-pdf/SP250-44.pdf) [3].

Determination of uncertainties

The basis for the determination of uncertainties associated with High-Dose calibrations is the Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results [4]. The purpose of this section is to explain the derivation of the various components of uncertainty for absorbed-dose certification. The values for the uncertainty components are listed in Appendix F.

Water Calorimetry: uncertainty from realization of the Gy [5].

Source Ratio Data: uncertainty from source dose-rate transfer (water calorimetry rate to high-dose calibration source rate) through ratio measurements.

Field Uniformity: radiation field uniformity within a dosimeter volume.

Environmental Effects: temperature control during irradiation.

Timer: uncertainty of timer readout relative to shortest irradiation time interval.

Decay Correction: half-life correction factor uncertainty.

Mass: uncertainty of microbalance relative to dosimeter mass.

Repeatability and Reproducibility: standard deviation of replicate dosimeter measurements.

Interspecimen Contamination: cross contamination of dosimeters during measurement process.

Ruby Correction: uncertainty resulting from EPR spectrometer fluctuations

during the time interval between the alanine dosimeter measurement and the reference (ruby) measurement.

System Drift: uncertainty arising from temporal EPR spectrometer response fluctuations.

Temperature Correction: uncertainty from alanine dosimeter temperature coefficient measurement.

Calibration Curve: fit uncertainty from alanine dosimeter calibration curve (See Appendix G).

References

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2. Radiation Processing Dosimetry Calibration Services: Manual of Calibration Procedures, Humphreys, J.C., Puhl, J.M., Seltzer, S.M., McLaughlin, W.L., Desrosiers, M.F., Bensen, D.L., Walker, M.L. 1998 NIST Special Publication 250-45.
3. Radiation Processing Dosimetry Calibration Services and Measurement Assurance Program, Humphreys, J.C., Puhl, J.M., Seltzer, S.M., McLaughlin, W.L., Desrosiers, M.F., Bensen, D.L., Walker, M.L. 1998 NIST Special Publication 250-44.
4. NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.
5. Domen, S.R., A sealed water calorimeter for measuring absorbed dose, J. Res. Natl. Inst. Stand. Technol., 99, pp. 121 – 141, 1994.

Records

Record	**Contents/Purpose**	**Location**
Calibration Log Book	Login all tests to obtain test folder number	C217
High-Dose Irradiations Databook	Records all dosimeter calibrations	C217
Irradiation Facilities Record Book	Records dose rates for irradiation geometries and instructions	B140
Dosimetry System Databook	Records dosimetry system calibrations and dosimeter batch characterization	C209
Internal Calibrations	Source ratio measurements and data analysis	C209
Quality Control	Check dose measurements and other routine quality control	C209
EMS104 Log Book	User and Maintenance records	C205

Record	Contents/Purpose	Location
ECS106 Maintenance Log Book	Maintenance records	C207
Transfer Dosimetry Databook	Records all transfer dosimeter certification data	C209
High-Dose International Comparisons Databook	Interlaboratory measurement comparisons data summaries	C217

Filing and Retention

All paper copies of customer files are stored in the test folder for that service. All customer-related electronic files are stored either on password-protected calibration-staff desktops or in the "High Dose" folder on the shared network drive.

The IRD Quality Manager shall maintain the original and all past versions of this IRD Procedure. Copies of the current revision of this Procedure shall be placed in controlled Quality Manuals. Electronic copies of this Procedure are uncontrolled versions.

All deleted Procedures (including old revisions) shall be maintained by the IRD Quality Manager. All old revisions shall be maintained until such time as it is decided to delete the Procedure. Once the decision has been made to delete the Procedure, only the last revision shall be maintained by the IRD Quality Manager.

Appendix A – Dosimeter Batch Acceptance Criteria

Dosimeter Test	Acceptance Level
Mass	≤ 10%
Height	≤ 10%
Dosimeter Response	≤ 1% (pellets)
	≤ 2% (film)
Fractionation	≤ 2% (pellets)
	≤ 4% (film)
Relative Humidity	≤ 3%
Time	≤ 2%
Temperature Coefficient	≤ 30%
Batch Response	≤ 20%

Appendix B – Sample Transfer Letter

September 3, 2003

HD0399

Cobalt Jones
Slamma Gamma Irradiators

Return Shipments to:
Sarenee L. Cooper
NIST High-Dose Service
Building 245, Room C229
100 Bureau Drive, Stop 8460
Gaithersburg , MD 20899-8460
USA

Dear Jason,

Enclosed are the alanine transfer dosimeters that you requested for irradiation in your facility. There are 6 vials for irradiation (9901-9906), each filled with three alanine pellets from the NIST batch A76. The other vial is a control and should not be irradiated. Do not open the vials. Please complete the following table and return a copy of this page with the returning dosimeters.

Dosimeter		**9901**	**9902**	**9903**
Date(s) of irradiation				
Target Dose, kGy (approximate)				
Average Temperature During Irradiation (deg.C)				
Dosimeter		**9904**	**9905**	**9906**
Date(s) of irradiation				
Target Dose, kGy (approximate)				
Average Temperature During Irradiation (deg.C)				
How would you like for us to identify your source on the report?				
Any other information you wish to be noted on the calibration report?				

Sincerely,

Sarenee L. Cooper
Calibrations Technician
Radiation Interactions & Dosimetry Group
Physics Laboratory
PHONE: 301-975-5054 FAX: 301-869-7682 E-MAIL: scooper@nist.gov

Enclosures

Appendix C – EPR Measurement Routine Programs

ECS106 Acquisition routine, Alaplusruby:

1> setp
2> wait 5 s
3> mtu
4> racq
5> tp inc 2
6> pg inc 1
7> setp
8> wait 5 s
9> mtu
10> racq
11> tp inc 2
12> pg inc 1
13> setp

Appendix D – QM Checklist for 49020C and 49030C

NIST ID:
Date:

Checklist for 49020C and 49030C:

____ The ECS106 Spectrometer was set up with the spectral parameters that were used for the corresponding calibration curve.
____ The spectral parameters were noted in the excel spreadsheet.
____ The pellet masses are paired correctly with the corresponding pellet number.
____ The excel spreadsheet reflects the appropriate NIST ID, company name, irradiation temperatures, etc.
____ The excel spreadsheet reflects the appropriate file names and dates for both the data file and calibration file.
____ The correct calibration curve coefficients were used in the dose calculation and noted on the spreadsheet.
____ All mathematical calculations embedded in cells have been checked for accuracy and correct cell linkage.
____ The appropriate correction factors were applied (i.e. temperature, dose-to-water, dose-silicon, Cs, etc.) to the calculated dose.

Signed by: _____ Date: _____

Appendix E – Example Certificate

National Institute of Standards and Technology

Absorbed-Dose Measurement Certificate

NIST Service Identification Numbers 49020C and 49030C

FOR
MDS Nordion Gammacell XXX

FOR
Slamma Gamma Irradiators
7 Electron Avenue
Mega Rad, LA 99817

ATTN: Cobalt Jones
Reference: PO # 5678

Measurements made by Sarenee L. Cooper

Report reviewed by Marc F. Desrosiers

Report approved by

Stephen M. Seltzer, Leader
Radiation Interactions and Dosimetry Group

For the Director
National Institute of Standards and Technology
By

Lisa R. Karam, Acting Chief
Ionizing Radiation Division
Physics Laboratory

Information on technical aspects of this report may be obtained from Sarenee L. Cooper, NIST, 100 Bureau Drive Stop 8460, Gaithersburg, MD 20899, 301-975-5054

REFERENCE HD0399
TEST FOLDER 123456
NIST DB 2000/155
October 8, 2003

Transfer dosimeters were sent to Slamma Gamma Irradiators for irradiation in their facility. The dosimeters were NIST alanine pellets of batch A76; three each in a polystyrene vial. Upon their return to NIST, the dosimeters were analyzed on September 23, 2003, using a Bruker ECS106 spectrometer. Dose interpolations are based on a NIST calibration of A76 alanine dosimeters performed May 21, 2003. The results are summarized in the following table.

Dosimeter Identification	Absorbed Dose kGy(H_2O)
9901	4.99
9902	4.98
9903	19.85
9904	19.69
9905	34.27
9906	34.62

UNCERTAINTIES AND RELATED FACTORS IN HIGH-DOSE MEASUREMENTS

Absorbed Dose Evaluations Based on Use of Mailed Alanine Pellet Transfer Standard Dosimeters Irradiated Using ^{60}Co

(Expanded uncertainty: ± 2.0% at a 95% confidence level)

The customer's use of NIST-certified transfer standard dosimeter measurements to determine their radiation-source dose rate is subject to limitations and precautions described in the letter accompanying the dosimeters. The customer must follow the prescribed procedures carefully in order to ensure that the results obtained from the transfer dosimeters are valid.

In the case of absorbed dose in water evaluation is based on NIST alanine pellet dosimeters that are traceable to standard water calorimeter measurements at NIST. The uncertainty value cited above may be assumed as long as suitable care is exercised. That value does not include uncertainty in the customer-reported irradiation temperature or non-uniformity in the customer's irradiation field.

A detailed list of the various sources of uncertainty and estimates of the magnitude of those uncertainties that make up the overall uncertainty given above may be obtained through the Internet (http://physics.nist.gov/Divisions/Div846/QualMan/index.html) or by requesting this information from NIST. The uncertainties are divided into two types: A and B. Type A uncertainties are those evaluated by statistical methods, often associated with random effects. Type B uncertainties are those evaluated by other means, often associated with systematic effects.

Type A Uncertainties

The combined standard uncertainty evaluated by statistical methods is ±0.65% at an approximate level of confidence of 68%.

Type B Uncertainties

The combined standard uncertainty based on scientific judgment is estimated to be ±0.76% at an approximate level of confidence of 68%.

Expanded Uncertainty

The type A and type B uncertainties have been combined in quadrature (the square root of the sum of the squares) and multiplied by a coverage t-factor of 2 to yield an expanded uncertainty of ±2.0% at an approximate level of confidence of 95%.

Appendix F- Uncertainties of Transfer Dosimetry Service

Domen Water Calorimeter B036, 11Jan1990

Uncertainty Source		Type A (%)	Type B (%)
Published values		0.16	0.32
Additional 5apr2001			0.40
	sqrt(sum)	0.16	0.51

GC207 Center Position Alanine Dose Rate

Uncertainty Source		Type A (%)	Type B (%)
Water Calorimetry in B036		0.16	0.51
GC207/Pool Source Ratio Data		0.29	
Pool/Vert. Beam Source Ratio Data		0.17	
Field uniformity			0.01
Temperature Correction			0.10
Timer Error (irrad time > 8min)			0.20
^{60}Co Decay Correction			0.02
	sqrt(sum)	0.37	0.56

High-Dose Alanine Response, Far West / Gamma Service Alanine Pellets

Uncertainty Source		Type A (%)	Type B (%)
Repeatability and Reproducibility		0.30	
Mass Determination		0.20	
Interspecimen contamination			0.10
Ruby correction			0.05
System Drift			0.10
	sqrt(sum)	0.36	0.15

Alanine Pellet Dosimeter Transfer Dose(water), Gamma/X-Ray, >200 Gy

Uncertainty Source		Type A (%)	Type B (%)
Alanine Dose Rate		0.37	0.56
Alanine Response		0.36	0.15
Temperature Correction			0.10
Dose Rate effect			0.10
Calibration Curve		0.50	0.10
	sqrt(sum)	0.72	0.60
	combined in quadrature		0.94
	t-factor for 50 d.f at 95.45%		2.05
	Expanded Uncertainty at 95.45% conf.		**1.9**

Alanine Pellet Dosimeter Transfer Dose(water), Gamma/X-Ray, ≤200 Gy

Uncertainty Source		Type A (%)	Type B (%)
Alanine Dose Rate		0.37	0.56
Alanine Response		0.36	0.15
Temperature Correction			0.10
Dose Rate effect			0.10
Calibration Curve		1.00	0.10
	sqrt(sum)	1.13	0.60
	combined in quadrature		1.28
	t-factor for 40 d.f at 95.45%		2.06
	Expanded Uncertainty at 95.45% conf.		**2.6**

Alanine Pellet Dosimeter Transfer Dose(silicon), Co-60

Uncertainty Source		Type A (%)	Type B (%)
Alanine Dose Rate		0.37	0.56
Alanine Response		0.36	0.15
Temperature Correction			0.10
Dose Rate effect			0.10
Calibration Curve		0.50	0.10
μ_{en} ratio water to silicon			1.80
	sqrt(sum)	0.72	1.90
	combined in quadrature		2.03
	t-factor for 50 d.f at 95.45%		2.05
	Expanded Uncertainty at 95.45% conf.		**4.2**

Alanine Pellet Dosimeter Transfer Dose(water), electron beam

Uncertainty Source		Type A (%)	Type B (%)
Alanine Dose Rate		0.37	0.56
Alanine Response		0.36	0.15
Temperature Correction			0.10
Dose Rate effect			0.10
Calibration Curve		0.50	0.10
Energy Dependence			0.10
	sqrt(sum)	0.72	0.61
	combined in quadrature		0.95
	t-factor for 50 d.f at 95.45%		2.05
	Expanded Uncertainty at 95.45% conf.		**1.9**

Alanine Pellet Dosimeter Transfer Dose(water), Cs-137

Uncertainty Source	Type A (%)	Type B (%)
Alanine Dose Rate	0.37	0.56
Alanine Response	0.36	0.15
Temperature Correction		0.10
Dose Rate effect		0.10
Calibration Curve	0.50	0.10
Cs-Co Correction	1.00	0.10
sqrt(sum)	1.23	0.61
combined in quadrature		1.38
t-factor for 20 d.f at 95.45%		2.13
Expanded Uncertainty at 95.45% conf.		**2.9**

High-Dose Alanine Response, Bruker/Kodak Alanine Films

Uncertainty Source	Type A (%)	Type B (%)
Repeatability and Reproducibility	1.10	
System Drift		0.10
sqrt(sum)	1.10	0.10

Alanine Film Dosimeter Transfer Dose(water), Gamma/X-Ray

Uncertainty Source	Type A (%)	Type B (%)
Alanine Dose Rate	0.37	0.56
Alanine Response	1.10	0.10
Temperature Correction		0.10
Dose Rate effect		0.10
Calibration Curve	0.90	0.10
sqrt(sum)	1.47	0.59
combined in quadrature		1.58
t-factor for 30 d.f at 95.45%		2.09
Expanded Uncertainty at 95.45% conf.		**3.3**

Alanine Film Dosimeter Transfer Dose(water), Electron Beam

Uncertainty Source	Type A (%)	Type B (%)
Alanine Dose Rate	0.37	0.56
Alanine Response	1.10	0.10
Temperature Correction		0.10
Dose Rate effect		0.10
Calibration Curve	0.90	0.10
Electron Energy Dependence		0.10
sqrt(sum)	1.47	0.60
combined in quadrature		1.59
t-factor for 30 d.f at 95.45%		2.09
Expanded Uncertainty at 95.45% conf.		**3.3**

Appendix G- Calibration Curve Fit Uncertainties Estimation

The following protocol is used to estimate uncertainties associated with the curve fit to calibration data. These protocols were developed in consultation with the NIST Statistical Engineering Division (SED). Our results were validated by comparing our computations to the NIST SED computations from identical data sets. All files cited here are located in the "High Dose" folder on the shared network drive.

For a linear function:

1. Open linear_fit_uncertainty.xls.
2. Follow instructions located there.

For non-linear functions:

1. Save x-y data as a text file.
2. Open appropriate ".boot" macro with NotePad (for macro details see below).
3. Edit read line to reflect text file name.
4. Save file.
5. Run DataPlot.
6. In DataPlot text window type "call abcd.boot" (where abcd is the selected macro name) and press enter.
7. DataPlot will write results as "abcd.out".
8. Open abcd.out in NotePad and select an appropriate uncertainty.

Code for cubic.boot:

```
dimension 40 columns
reset data
. ..device 1 x11
. ..
skip 1
read kodak307data.txt x y
. ..
let n = number y
let minx = minimum x
let maxx = maximum x
let minx = minx - 2
let maxx = maxx + 2
. ..
cubic fit y x
let ycalib = distinct pred
let ncalib = size ycalib
.
.  Check fit first
.
```

```
line bl so
char x bl
plot y pred vs x
.
let x1 = x
let x2 = x*x
let x3 = x*x*x
bootstrap samples 200
bootstrap fit y x1 x2 x3
skip 0
read dpst1f.dat a0coef a1coef a2coef a3coef
system cp dpst1f.dat bootstrap_coef.txt
let nboot = size a0coef
. ..
. .. Now loop to perform the calibrations
. ..
feedback off
printing off
.
loop for k = 1 1 ncalib
  let y0 = ycalib(k)
  loop for l = 1 1 nboot
    let a0 = a0coef(l)
    let a1 = a1coef(l)
    let a2 = a2coef(l)
    let a3 = a3coef(l)
    let function f1 = a3*x**3 + a2*x**2 + a1*x + a0 - y0
    let r = roots f1 wrt x for x = minx maxx
    let rr = r(1)
    let xtemp(l) = rr
    let xtag(l) = k
 end of loop
 if k = 1
   let x0 = xtemp
   let tag = xtag
 else
   extend x0 xtemp
   extend tag xtag
 end of if
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
```

```
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write kodak307cubic.out ycalib mb sb rb
. ..
```

Code for katz2term.boot:

```
. ..
. ..  sample bootstrap code for
. ..  calibration interval estimate
. ..  on EXP model inversion
. ..
dimension 20 columns
. ..
skip 1
read marc.nonlinear x y
let n = number y
. ..
fit y = a*(1-exp((-x)/b))
let astart = a
let bstart = b
. ..
let pred2 = pred
let y0 = distinct pred2
let ndist = size y0
let xtag = sequence 1 1 ndist
let res2 = res
let numboot = 200
. ..
let a = astart
let b = bstart
let ind = bootstrap index for i = 1 1 n
let res3 = bootstrap sample res2 ind
let y3 = pred2+res3
fit y3 = a*(1-exp((-x)/b))
let x0 = (-b)*log(1-(y0/a))
let tag = xtag
. ..
loop for k = 2 1 numboot
   let a = astart
   let b = bstart
   let ind = bootstrap index for i = 1 1 n
```

```
   let res3 = bootstrap sample res2 ind
   let y3 = pred2+res3
   fit y3 = a*(1-exp((-x)/b))
   let xtemp = (-b)*log(1-(y0/a))
   extend tag xtag
   extend x0  xtemp
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write katz2term.out y0 mb sb rb
. ..
. .. exit
```

Code for katz3term.boot:

```
. ..
. ..  sample bootstrap code for
. ..  calibration interval estimate
. ..  on EXP model inversion
. ..
dimension 20 columns
. ..
reset data
skip 1
read gs604datald.txt x y
let n = number y
. ..
let function f = a*(1-exp(-(x+b)/c))
pre-fit y = f for a = 50 25 500 for b = .01 0.05 .1 for c = 25 25 150
fit y = f
let astart = a
let bstart = b
let cstart = c
. ..
let pred2 = pred
let y0 = distinct pred2
let ndist = size y0
let xtag = sequence 1 1 ndist
```

```
let res2 = res
let numboot = 200
. ..
let a = astart
let b = bstart
let c = cstart
let ind = bootstrap index for i = 1 1 n
let res3 = bootstrap sample res2 ind
let y3 = pred2+res3
fit y3 = a*(1-exp(-(x+b)/c))
let x0 = -b-(c*log(-(y0-a)/a))
let tag = xtag
. ..
loop for k = 2 1 numboot
   let a = astart
   let b = bstart
   let c = cstart
   let ind = bootstrap index for i = 1 1 n
   let res3 = bootstrap sample res2 ind
   let y3 = pred2+res3
   fit y3 = a*(1-exp(-(x+b)/c))
   let xtemp = -b-(c*log(-(y0-a)/a))
   extend tag xtag
   extend x0  xtemp
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write gs604ld.out y0 mb sb rb
. ..
line bl so
char x bl
plot y0 vs mb
. ..
```